Final Report

on

POTASSIUM VAPOUR IMPREGNATION OF METALLURGICAL COKE

Contract File No. 20ST.23440-5-9157

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to be submitted to

Supply and Services Canada

and

CANMET, EMR

Scientific Authority of the Contract: Dr. John T. Price
CANMET

by

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MICROMEDIA

SUMMARY

A new experimental technique has been developed in the present work to have coke lumps (20 mm) impregnated with potassium in precise amounts in K-Ar mixtures. Potassium impreghation of each lump (all specimens were pre-heated for de-volatilization) was monitored by weight gain. The measurements of weight gain for chemical analysis have been confirmed by neutron activation analysis in two laboratories to be acceptable. Treated coke lumps of known amounts of potassium were forwarded to Dr. John T. Price to be used in the CSR tests.

In addition to the contractual work as stated above, efforts have been made to identify the cause of the variation in the kinetics of potassium absorption from lump to lump, as partly summarized in Tables 5 through 14. Because of the limited time devoted to this investigation, the results are not conclusive at the present time; however, future work has been outlined and presented.

1. INTRODUCTION

As a small part of the overall program of characterization and evaluation of metallurgical cokes at CANMET, EMR this contract is concerned with a special treatment of samples before CSR tests. Our service is to have coke samples which are prepared for CSR (Coke Strength after Reaction) tests, to absorb fixed amounts of potassium (0.2% and 0.5% wt., respectively) from a binary mixture, de-oxidized argon and metallic vapour of potassium, at 1100°C. It was intended to simulate this very important feature of actual blast furnace conditions and to investigate the effect of potassium in CSR tests. Nine different types of coke were investigated. Coke lumps 20 mm in size, prepared from cokes made in CANMET's pilot plant, were provided by Dr. John T. Price, the scientific authority of the contract.

2. SOLUTIONS TO EXPERIMENTAL DIFFICULTIES

A. Accurate Measurement of the Amount of Potassium Absorbed

It has to be non-destructive. It has been reported in the literature that the general practice is to have a sample of several hundred grams of coke treated in a single run. The overall weight gain is considered to be the amount of potassium absorbed. In order to assess the uniformity of potassium absorption, in these preliminary experiments samples each weighing 65 grams were used in each run and every piece of coke in the sample was weighed separately to determine its weight gain. The variations of weight gain, (supposedly the amount of potassium absorbed by each piece of coke) were too large to be acceptable.

It was recognized that during the treatment of potassium impregnation at 1100°C de-volatilization which results in weight loss, took place simultaneously. The extent of de-volatilization depends on coal blend, coking temperature and the location of individual coke pieces in the oven when carbonization took place, etc. A pre-treatment of coke samples prior to potassium impregnation at 1150°C for 30 minutes under flowing de-oxidized argon was established. The size of coke sample in each run was decreased from 65 grams to 25 grams so that the few pieces of coke located downstream would not contact with gas of significantly lower potassium content. Under these conditions, results obtained in potassium impregnation are documented in this report.

The amount of potassium absorbed by weight gain of coke pieces has also been checked by another method, i.e., neutron activation analysis.

B. Hitting the Targets of 0.2 and 0.5 wt.% Weight-Gain

The temperature of impregnation was kept constant in all experiments. The vapour pressure was maintained in a narrow range. In the preliminary experiments an empirical relationship between the time of reaction and the amount of potassium absorbed for each coke was obtained. By controlling the reaction time, the desired amount of potassium was impregnated in each case.

3. EXPERIMENTAL DESIGN AND PROCEDURES

A. Experimental Design

The apparatus designed to provide required conditions is shown schematically in Figure 1. A 2" I.D. single horizontal Inconel tube with sealed ends was chosen, with two independent heating system electric furnaces. Electric Furnace II provides potassium vapour by heating up the metallic melt which was in the stainless steel vessel. Electric Furnace I maintains the reaction zone at desired temperature. The temperature inside the system was closely monitored by chromel-alumel thermocouples, placed at the edge of either the vessel containing the metallic melt, or the container of coke samples. Deoxidized argon as a carrying gas has been used. Alumina balls were placed at the inlet side and also heating tape (350°C) and thermal insulation on the inlet side to provide heating and mixing. The shield (stainless steel tube) of thermocouple (TI) was designed with O-rings sealing and also moveable to serve as the control rod to move the samples between the cool zone near the gas outlet and the heat zone.

(B) Experimental Procedures

All samples were heat treated before being used for impregnation experiments. After the weighing of each lump of coke the samples were placed in a sample holder made from stainless steel wire mesh. The vessel with the samples was introduced to the system from the exit end of the gas and placed inside the tube at the "cold zone" at 150°C, followed by the sealing of the system and argon purging of the system for 5 minutes at a flow rate of 0.5 l/min. The next step was sliding the sample holder to the hot zone by thermocouple shield (see Fig. 2). It took 12 minutes to reach treatment temperature from 150°C to 1100°C. During this time the flow rate of the argon was 1 ml/sec (20 psi). After the samples had reached the required temperature of 1100°C, a potassium vapour was introduced by heating up argon (2.0 l/min at 20 psi) and potassium melt (220 - 225°C) to result in a partial pressure of potassium estimated to be in the range of 10^{-4} to 10^{-3} atm. Then the samples were left for a pre-determined time either to reach 0.2% or 0.5% weight gain. At the end of the run the sample was gradually pulled out from the hot zone to the cold zone, and cooled down to 150°C in 12 minutes. During this period of the experiments the flow rate of the gas was lowered to 1 ml/sec (20 psi) and followed by placing the samples in a dessicator to cool down to room temperature. Then each piece of coke lump was weighed.

4. RESULTS

A. Heat Treatment Before Potassium Impregnation

For each of the nine types of coke about 20 specimens were heat treated at 1150°C for 30 minutes in de-oxidized argon; the results are listed in Table 1. It is very clear that such treatment is necessary in order to measure at weight gain of 0.2% and 0.5%.

B. Potassium Impregnation

In Table 2, the overall situations are presented. For the impregnation experiments aimed at 0.2% weight gain the number of coke lumps treated is in the range from 72 to 114. For example, 104 pieces of coke lumps CPR296 were treated to result in an average 0.22% weight gain, with standard deviation 0.1%. However, 44 pieces were selected for the contract, i.e., shipped to CANMET for CSR tests, to result in an average weight gain of 0.20% and a standard deviation of 0.025%. In general, for 0.2% weight gain the standard deviation is less than 0.04% and that for 0.5% weight gain is less than 0.07%. For clarity, data which respresents prepared samples that have been shipped to CANMET, are listed in Table 3.

C. Chemical Analysis

Various methods of measurement of potassium absorption are listed in Table 4. The values obtained by weight gain and by neutron activation analysis agree quite well with a small but systematic difference; see Fig. 3. The results based on atomic absorption are not acceptable because there was an error in the preparation of the solution.

5. DISCUSSIONS

A. The Effect of Pre-Treatment at 1150°C

In the present work, pre-treatment at 1150°C for 30 minutes was done to promote de-volatilization so that it would not cause significant error at 1100°C during subsequent potassium impregnation. It is well known that final carbonization temperature in cokemaking influences the characteristics of coke, i.e., chemical reactivity and CSR values. In case the differences in coking conditions, particularly temperature, are major variables of study, the pretreatment at 1150°C would reduce the clarity of the signals which to be observed. However, for CSR tests in which the gasification already takes place at 1100°C anyway, the thermal effects on specimens at 1150°C for pre-treatment and 1100°C for impregnation may be overshadowed by the effect of potassium.

B. The Effect of Positions of Individual Specimens in Relation to the Gaseous Flow During Potassium Impregnation

One obvious concern of ours is whether or not the variation of the amount of potassium absorbed by different specimens in a run is related to the diminishing potassium content in the gas as it flows further downstream. As shown in Fig. 2, specimens were placed in three rows facing the flow of gas. Among all the data in our possession, two types of experimental results are listed in Tables 5 to 13. They are selected for the situations that specimens in the second row are either smaller or larger in comparison with the other four in the same run. In the first three columns of the table the average value (of two specimens) of each row are listed respectively. In the fourth and fifth columns of each table the differences in weight gain between the first two rows and that between the last two rows in the same experiment are listed, respectively. Except for CPR307, CPR308 and to a certain extent, CPR300 which showed some noticeable abnormality (in the view of standard deviation of data) specimens placed in

the first row absorbed more potassium than those placed in the downstream of gas flow.

C. Experiments With Much Reduced Sample Size

The results listed in Tables 5 to 13 as discussed in Section B above, strongly suggested that the diminishing potassium content in the gas is likely to be the cause. A series of experiments was carried out with only three lumps of coke instead of six to reduce the possible "starvation effect" in potassium impregnation. Experimental results of five types of coke, including CPR307 and CPR308, are listed in Table 14. The variation in potassium asborption appears to be random. The collection of data is too small to be treated statistically.

D. Distribution of Potassium in Each Coke Lump

The distribution of "degree of burn-off" in a partially gasified specimen by oxidation is often closely related to the distribution of potassium (and/or sodium). For the same degree of burn-off its distribution has a pronounced effect on the weakening of its mechanical strength. In our prepared specimens, we have no knowledge of the distribution of potassium.

E. Susceptibility to Potassium Attack of Cokes in Blast Furnaces

If the assumption is accepted that more potassium in coke is worse for its performance in blast furnaces is accepted, there is hope that such susceptibility might be defined. From Table 14, it is reasonable to conclude that the dominant factor which causes the variations in potassium impregnation has to be a property (or a combination of properties) of coke. It would be extremely interesting for academic as well as practical implications to identify this property of coke.

6. CONCLUSION

A. All contractual responsibilities have been successfully fulfilled.

B. In addition to work specified in the contract, kinetic studies relevant to the overall objectives have been carried out.

C. As outlined below, future work is recommended to clarify the susceptibility of coke to potassium attack in blast furnaces.

7. RECOMMENDED FUTURE WORK

A. Reactivity of Powder Coke

With financial support from CANMET, NSERC and Imperial Oil of Canada, the research team under the supervision of this writer, has conducted investigations to correlate reactivity with respect to CO_2 oxidation and properties of coke. Porosity, specific surface and texture of carbon grains have been investigated. Correlations between reactivity and three coke properties mentioned above are inconclusive.

Kinetics of moisture absorption by coke powders in a dessicator which was maintained at constant temperature (25 to 35°C) and with a beaker containing water have been studied at McMaster University. The rate of moisture absorption has been found to be proportional to reactivity. It is due to the fact that the rate of moisture absorption is related to the amount of chemically reactive pore surface (excluding the very fine pores) and the connectivity of pores.

B. Non-Destructive Test of Reactivity of Coke Lumps

The relationship between the rate of moisture pick-up by coke lumps of 20 mm size and reactivity with respect to CO_2 may be established. From a given coke, lumps may be separated based on the rate of moisture absorption. Batches of coke separated in this manner may be evaluated by CSR tests.

C. Non-Destructive Test of Susceptibility of Coke to Potassium Attack

Investigations outlined in Section B above may be repeated except that the CSR test would be replaced by potassium impregnation.

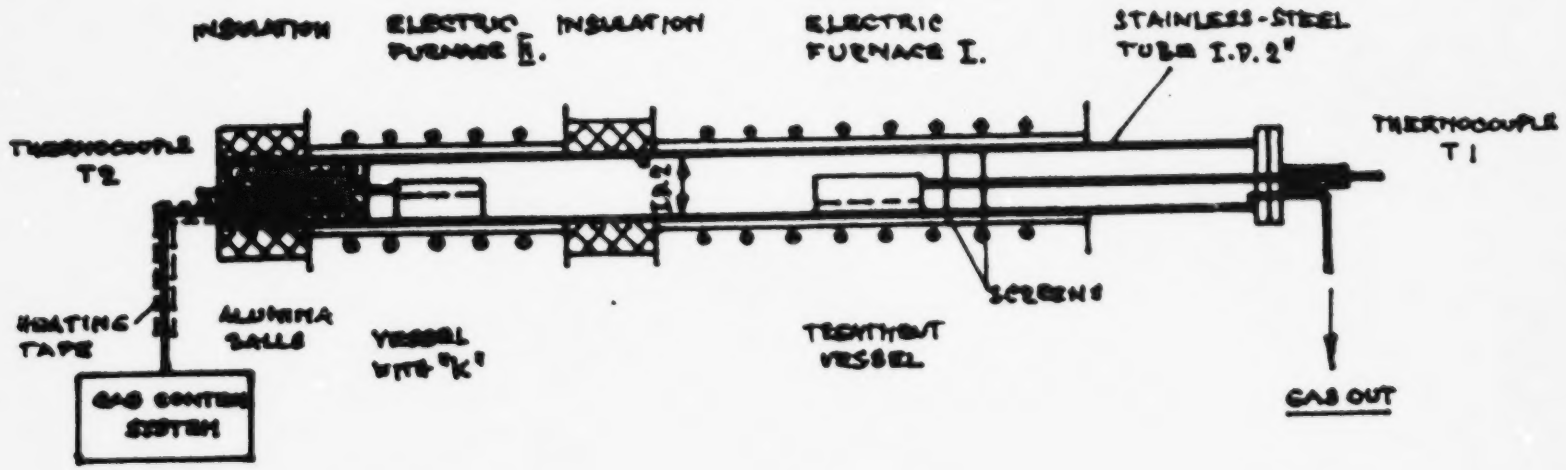
INSULATION
ELECTRIC FURNACE II.
INSULATION
ELECTRIC FURNACE I.
STAINLESS-STEEL TUBE I.D. 2"
THERMOCOUPLE T2
THERMOCOUPLE T1
HEATING TAPE
ALUMINA BALLS
VESSEL WITH "K"
TEFLON VESSEL
SCREENS
GAS OUT
GAS CONTROL SYSTEM

FIGURE 1.

FIGURE 2

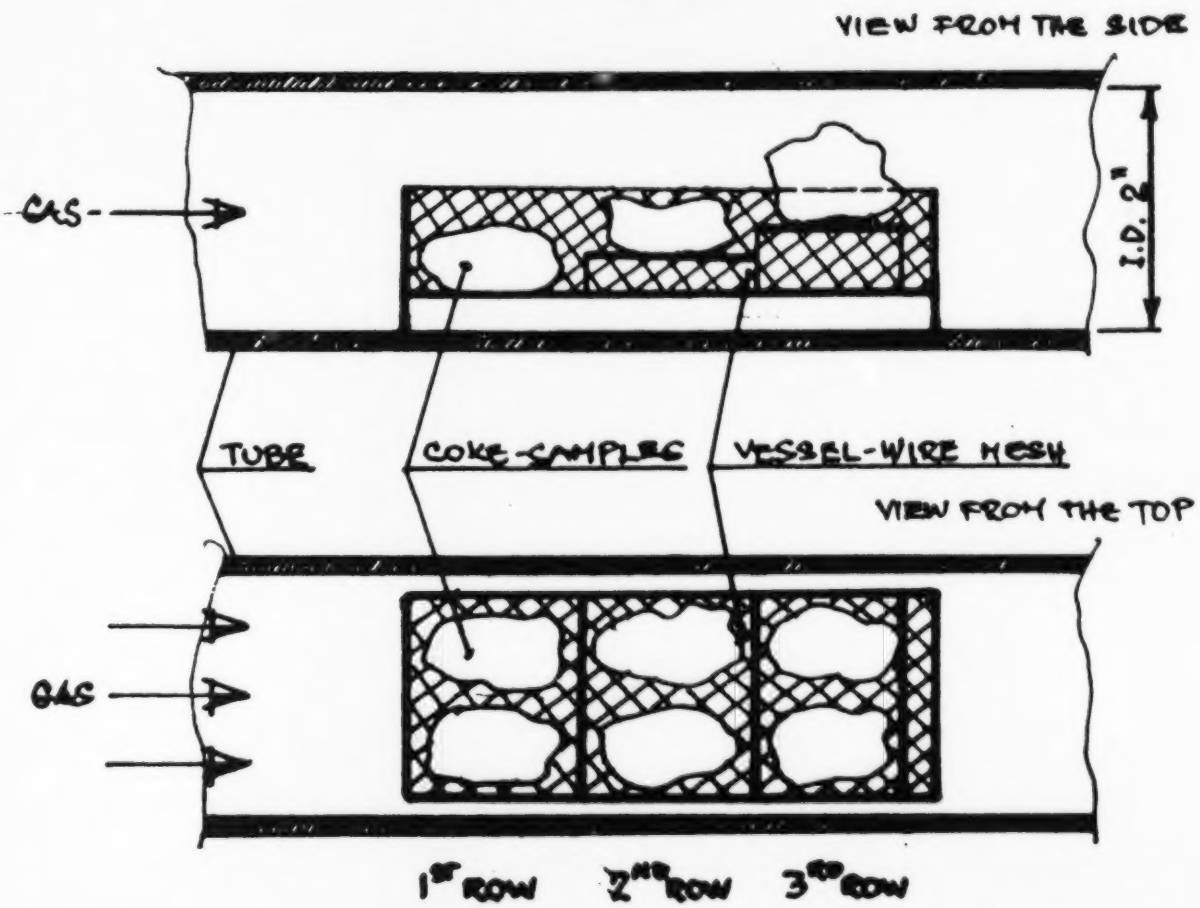
VIEW FROM THE SIDE
GAS
I.D. 2"
TUBE
COKE-SAMPLE
VESSEL-WIRE MESH
VIEW FROM THE TOP
GAS
1ST ROW
2ND ROW
3RD ROW

FIGURE 3

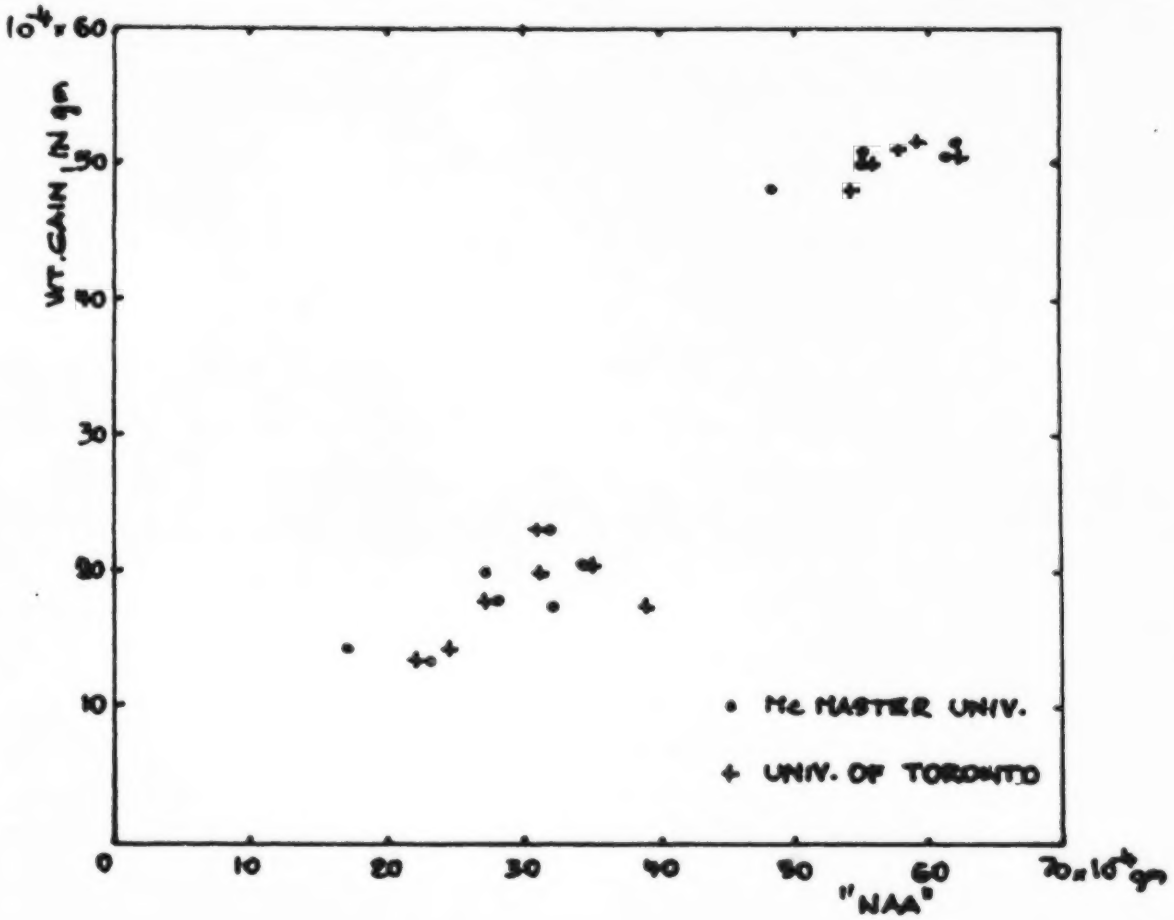
10^{-4} x 60
WT. GAIN IN gm
50
40
30
20
10
0
10
20
30
40
50
60
20×10^{-4} gm
"NAA"
• Mc MASTER UNIV.
+ UNIV. OF TORONTO

TABLE 1

COKE	NUMBER OF SAMPLES	HEAT TREATED AT 1150°C/30 MIN. Ar GAS WEIGHT LOST, IN %			
		HIGH	LOW	$\bar{x}$	σ_n
CPR 296	19	0.60	0.20	0.36	0.10
CPR 299	23	1.00	0.20	0.46	0.20
CPR 300	24	0.60	0.04	0.27	0.10
CPR 301	24	0.60	0.16	0.32	0.10
CPR 303	24	0.50	0.17	0.24	0.10
CPR 305	24	0.60	0.17	0.28	0.10
CPR 306	24	0.50	0.19	0.28	0.10
CPR 307	24	0.60	0.18	0.33	0.10
CPR 308	24	0.70	0.17	0.34	0.10

TABLE 2

COKE	0.2% WT. GAIN RANGE						0.2% WT. GAIN RANGE					
	OVERALL			SELECTED			OVERALL			SELECTED		
	No. of P_{cs}.	$\bar{x}$	σ_n	No. of P_{cs}.	$\bar{x}$	σ_n	No. of P_{cs}.	$\bar{x}$	σ_n	No. of P_{cs}.	$\bar{x}$	σ_n
CPR 296	104	0.22	0.10	44	0.20	0.03	74	0.45	0.14	45	0.49	0.04
CPR 299	72	0.22	0.08	46	0.19	0.03	83	0.45	0.15	43	0.49	0.03
CPR 300	92	0.20	0.07	60	0.19	0.03	86	0.45	0.13	59	0.47	0.03
CPR 301	103	0.23	0.13	56	0.20	0.04	124	0.47	0.19	65	0.48	0.06
CPR 303	83	0.23	0.10	57	0.18	0.04	93	0.45	0.14	58	0.47	0.06
CPR 305	114	0.19	0.06	63	0.18	0.04	127	0.58	0.30	64	0.47	0.06
CPR 306	93	0.21	0.06	57	0.21	0.04	80	0.51	0.30	54	0.47	0.06
CPR 307	91	0.19	0.07	64	0.19	0.04	135	0.62	0.40	70	0.48	0.07
CPR 308	107	0.19	0.07	48	0.18	0.03	82	0.52	0.20	53	0.48	0.06

TABLE 3

COKE	IMPREGNATED AT 1100°C ARGON GAS, P_K 10^{-3}		
	$\bar{x}$	σ_n	Total Wt. gm
CPR 296	0.20	0.03	200.6 + 25
	0.49	0.04	200.9 + 25
CPR 299	0.19	0.03	200.4 + 21.3
	0.49	0.03	201.4 + 24.7
CPR300	0.19	0.03	229
	0.47	0.03	228.5
CPR 301	0.20	0.04	225.5
	0.48	0.06	225.7
CPR 303	0.19	0.04	224.3
	0.47	0.05	230
CPR 305	0.18	0.04	221
	0.47	0.06	225.8
CPR 306	0.21	0.04	221
	0.47	0.06	226.7
CPR 307	0.19	0.04	225
	0.48	0.07	225
CPR 308	0.18	0.03	226
	0.48	0.06	227.8

TABLE 4

SAMPLE	K-CONTENT WT. GAIN	K-CONTENT NEUTRON ACTIVATION ANALYSIS		K-CONTENT ATOMIC ABSORPTION ANALYSIS
		McMaster University	University of Toronto	
REPUBLIC				
8-13	2.78 gm x 10^{-2}	2.3 gm x 10^{-2}		
8-14	2.51	2.5		
8-15	1.28	1.6		
8-16	2.49	2.2		
8-17	1.71	2.7		
8-18	2.38	1.7		
10-38	3.53	4.7		
10-39	5.07	5.2		
10-40	3.54	3.0		
10-41	1.88	2.3		
10-42	2.84	2.3		
10-43	3.57	2.7		
CPR 296- 1	5.06 gm x 10^{-3}	6.1 gm x 10^{-3}	6.2 gm x 10-3	8.1 gm x 10^{-6}
2	5.13	6.2	5.9	7.1
18	5.005	5.5	5.5	6.6
38	5.043	5.5	5.8	3.1
40	4.875	4.8	5.4	6.5
8	2.06	3.4	3.4	4.6
10	1.752	3.2	3.9	3.2
33	2.034	2.7	3.2	2.9
35	1.789	2.8	2.7	2.2
41	2.29	3.2	3.1	2.4
50	1.343	8.3	2.2	1.9
51	1.467	1.7	2.4	2.1

TABLE 5

Small Specimen in the Second Row					Large Specimen in the Second Row				
Wt. Gain, In %			$2^R - 1^{Rx}$*	$3^R - 2^R$	Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$
1st Row	2nd Row	3rd Row			1st Row	2nd Row	3rd Row		
0.50 0.50	0.46 0.46	0.30	−0.04 −0.04	−0.16	0.40 0.20	0.20 0.20	0.20 0.10	−0.20 0	0 −0.10
0.45 0.47	0.30 0.20	0.46 0.30	−0.15 −0.27	+0.16 +0.10	0.56 0.70	0.50 0.30	0.50 0.20	−0.06 −0.40	0 −0.10
0.50 0.70	0.30	0.30 0.30	−0.20	0	0.20 0.20	0.10 0.10	0.10 0.10	−0.10 −0.10	0 0
0.56 0.45	0.45 0.20	0.30 0.20	−0.11 −0.25	−0.15 0	0.30 0.30	0.20 0.20	0.13 0.20	−0.10 −0.10	−0.07 0
0.20 0.20	0.20 0.30	0.10 0.15	0 +0.10	−0.10 −0.15	0.49 0.30	0.10 0.20	0.20 0.20	−0.39 −0.10	+0.10 0
0.20 0.20	0.20 0.13	0.14 0.10	0 −0.07	−0.06 −0.03	0.52 0.54	0.35	0.32	−0.17	−0.03
0.15 0.36	0.12 0.26	0.12 0.12	−0.03 −0.10	0 −0.14	0.48 0.40	0.30 0.37	0.23 0.16	−0.18 −0.03	−0.07 −0.21
0.25 0.36	0.17 0.20	0.17 0.20	−0.08 −0.16	0 0	0.33 0.40	0.16 0.30	0.18 0.20	−0.17 −0.10	+0.02 −0.10
0.21 0.22	0.16 0.20	0.14	−0.05 −0.02	−0.06	0.12 0.17	0.16 0.16		+0.04 −0.01	
0.47 0.46	0.31 0.26	0.17	−0.16 −0.20	−0.14	0.57 0.65	0.37 0.56	0.26 0.37	−0.20 −0.09	−0.11 −0.19
0.20 0.20	0.20 0.23	0.20 0.19	0 +0.03	0 −0.04	0.28	0.36 0.27	0.29 0.24	+0.08	−0.07 −0.03
0.17 0.20	0.12 0.10	0.10 0.20	−0.05 −0.10	−0.02 +0.10	0.78	0.47 0.54	0.41 0.36	−0.31	−0.06 0.18
0.33	0.18 0.24	0.15 0.21	−0.15	−0.03 −0.03	0.38 0.26	0.24 0.23	0.26 0.18	−0.14 −0.03	+0.02 −0.05
0.73	0.51 0.39	0.31 0.28	−0.22	−0.20 −0.11	0.83	0.49 0.48	0.46 0.25	−0.34	−0.03 −0.23
0.55	0.66 0.45	0.37 0.30	+0.11	−0.29 −0.15	0.21	0.13 0.17	0.16 0.15	−0.08	+0.03 −0.02
0.56	0.47 0.48	0.26 0.40	−0.09	−0.21 −0.08					
0.60 0.55	0.46 0.47	0.32 0.34	−0.14 −0.08	−0.14 −0.13					
0.69 0.78	0.47 0.56	0.45 0.50	−0.22 −0.22	−0.02 −0.06					
0.46	0.45 0.46	0.46 0.47	−0.01	+0.01 +0.01					
0.26 0.10	0.15 0.23	0.20 0.25	−0.11 +0.13	+0.05 +0.02					
0.20 0.19	0.19 0.15	0.23	−0.01 −0.04	+0.04					

* 1^{Rx}: The data in the column under first row on the left of this table.

COKE CPR 299

TABLE 6

Small Specimen in the Second Row					Large Specimen in the Second Row				
Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$	Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$
1st Row	2nd Row	3rd Row			1st Row	2nd Row	3rd Row		
0.22	0.17 0.19	0.19 0.19	−0.05	+0.02 0	0.15 0.14	0.11 0.10	0.11 0.10	−0.04 −0.04	0 0
0.19	0.18 0.19	0.18 0.20	−0.01	0 +0.01	0.51 0.48	0.48 0.36	0.50 0.37	−0.03 −0.12	+0.02 +0.01
0.78	0.46 0.38	0.48 0.35	−0.32	+0.02 −0.03	0.65 0.52	0.34 0.50	0.35 0.46	−0.31 −0.02	+0.01 −0.04
0.54	0.40 0.46	0.37 0.47	−0.14	−0.03 +0.01	0.20 0.23	0.10 0.19	0.20 0.17	−0.10 −0.04	+0.10 −0.02
0.75	0.48 0.72	0.54 0.52	−0.27	+0.06 −0.20	0.29 0.24	0.14 0.17	0.16 0.21	−0.15 −0.07	+0.02 +0.04
0.75	0.48 0.82	0.68 0.53	−0.27	+0.20 −0.29	0.33 0.21	0.18 0.18	0.23 0.19	−0.15 −0.03	+0.05 +0.01
0.48 0.47	0.46 0.36	0.23 0.47	−0.02 −0.11	−0.23 +0.11	0.52 0.50	0.30 0.46	0.47 0.54	−0.22 −0.04	+0.17 +0.08
0.28	0.19 0.18	0.20 0.22	−0.09	+0.01 +0.04	0.55 0.96	0.48 0.53	0.61 0.26	−0.07 −0.43	+0.13 −0.27
0.49 0.47	0.31 0.46	0.49 0.30	−0.18 −0.01	+0.18 −0.16	0.49 0.41	0.26 0.31	0.27 0.33	−0.23 −0.10	+0.01 +0.02
0.19 0.30	0.17 0.23	0.11 0.19	−0.02 −0.07	−0.06 −0.04	0.41 0.35	0.26 0.30	0.29 0.38	−0.15 −0.05	+0.03 +0.08
0.24 0.19	0.15 0.16	0.23 0.15	−0.09 −0.03	+0.08 −0.01					
0.49	0.21 0.27	0.26 0.27	−0.28	+0.05 0					
0.37	0.25 0.33	0.28 0.29	−0.12	+0.03 −0.04					
0.48	0.22 0.33	0.26 0.27	−0.26	+0.04 −0.06					
0.46 0.53	0.31 0.46	0.47 0.26	−0.15 −0.09	+0.16 −0.20					
0.30 0.53	0.20' 0.27	0.20 0.23	−0.10 −0.26	0 −0.04					
0.30 0.35	0.24 0.33	0.29 0.26	−0.06 −0.02	+0.05 −0.08					

COKE CPR 300

TABLE 7

Small Specimen in the Second Row					Large Specimen in the Second Row				
Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$	Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$
1st Row	2nd Row	3rd Row			1st Row	2nd Row	3rd Row		
0.19 0.20	0.15 0.10	0.13 0.16	−0.04 −0.10	−0.02 +0.06	0.42	0.27 0.37	0.45 0.38	−0.15	+0.18 +0.01
0.24 0.24	0.11 0.19	0.15 0.18	−0.13 −0.05	+0.04 −0.01	0.13 0.35	0.24 0.34	0.30 0.44	+0.11 −0.01	+0.06 +0.10
0.14 0.17	0.10 0.10	0.10 0.15	−0.04 −0.07	0 +0.5	0.21 0.31	0.12 0.17	0.16 0.11	−0.09 −0.14	+0.04 −0.06
0.32 0.26	0.31 0.32	0.36 0.34	−0.01 +0.06	+0.05 +0.02	0.16 0.27	0.14 0.11	0.16 0.20	−0.02 −0.16	+0.02 +0.09
0.16 0.21	0.14 0.13	0.11	−0.02 −0.08	−0.03	0.18 0.24	0.10 0.17	0.10 0.18	−0.08 −0.07	0 +0.01
0.21 0.41	0.20	0.24 0.35	−0.21	+0.15	0.49 0.55	0.46 0.47	0.35 0.40	−0.03 −0.08	−0.11 −0.07
0.33 0.39	0.25 0.36	0.25 0.31	−0.08 −0.03	0 −0.05	0.22 0.27	0.11 0.22	0.20 0.28	−0.11 −0.05	+0.09 +0.06
0.28	0.19 0.22	0.23 0.23	−0.09	+0.04 +0.01	0.64 0.65	0.42 0.49	0.32 0.44	−0.22 −0.16	−0.10 −0.05
0.38 0.24	0.23 0.22	0.20 0.30	−0.15 −0.02	−0.03 +0.08	0.43 0.40	0.26 0.28	0.29 0.40	−0.17 −0.12	+0.03 +0.12
0.29 0.35	0.20 0.23	0.34 0.19	−0.09 −0.12	+0.14 −0.04	0.25 0.31	0.13 0.20	0.12 0.31	−0.12 −0.11	−0.01 +0.11
					0.39 0.35	0.22 0.32	0.23 .40	−0.17 −0.03	+0.01 +0.08
					0.25 0.27	0.18 0.11	0.24 0.17	−0.07 −0.16	+0.06 +0.06
					0.18 0.22	0.10 0.15	0.14 0.17	−0.08 −0.07	+0.04 +0.02
					0.22 0.19	0.10 0.15	0.14 0.17	−0.12 −0.04	+0.04 +0.02

COKE CPR 301

TABLE 8

Small Specimen in the Second Row					Large Specimen in the Second Row				
Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$	Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$
1st Row	2nd Row	3rd Row			1st Row	2nd Row	3rd Row		
0.13 0.17	0.10 0.10	0.04 0.05	−0.03 −0.07	−0.06 −0.05	0.40 0.38	0.22 0.27	0.25 0.31	−0.18 −0.11	+0.03 +0.04
0.27 0.17	0.10 0.13	0.08 0.07	−0.17 −0.04	−0.02 −0.06	0.23 0.28	0.10 0.22	0.26 0.22	−0.13 −0.06	+0.16 0
0.22 0.23	0.18 0.14	0.13 0.16	−0.04 −0.09	−0.06 +0.02	0.29 0.28	0.14 0.17	0.18	−0.15 −0.11	+0.04
0.21 0.34	0.10 0.22	0.10 0.15	−0.11 −0.12	0 −0.07	0.18 0.18	0.10 0.12	0.10	−0.08 −0.06	0
0.45	0.20 0.25	0.20 0.16	−0.25	0 −0.09	0.37 0.43	0.22 0.27	0.21 0.15	−0.15 −0.16	−0.01 −0.12
0.20 0.24	0.10 0.10	0.15 0.10	−0.10 −0.14	+0.05 0	0.58 0.41	0.23 0.20	0.18 0.27	−0.35 −0.21	−0.05 +0.07
0.14 0.15	0.10 0.12	0.10 0.10	−0.04 −0.03	0 −0.02	0.31 0.35	0.20 0.26	0.21 0.22	−0.11 −0.09	+0.01 −0.04
0.61 0.63	0.42 0.36	0.32 0.38	−0.19 −0.29	−0.10 +0.02	0.29 0.36	0.10 0.18	0.18 0.10	−0.19 −0.18	+0.08 −0.08
0.69 0.63	0.40 0.56	0.30 0.50	−0.29 −0.07	−0.10 −0.06	0.26 0.24	0.18 0.19	0.14 0.25	−0.08 −0.05	−0.04 +0.06
0.52	0.37 0.40	0.48 0.59	−0.15	+0.11 +0.19	0.12 0.17	0.10 0.10	0.10 0.10	−0.02 −0.07	0 0
0.54 0.65	0.30 0.44	0.40 0.40	−0.24 −0.21	+0.10 −0.04	0.17 0.18	0.12 0.11	0.12 0.10	−0.05 −0.07	0 −0.01
0.38 0.57	0.33 0.32	0.24 0.52	−0.05 −0.25	−0.09 +0.20	0.39	0.34 0.4	0.51 0.67	−0.05	+0.17 +0.27
0.62 0.89	0.46 0.38	0.24 0.24	−0.16 −0.51	−0.22 −0.14	0.64 0.62	0.31 0.41	0.34 0.47	−0.33 −0.21	+0.03 +0.06
0.44	0.46 0.45	0.37 0.47	+0.02	−0.09 +0.02	0.84 0.67	0.40 0.38	0.35 0.57	−0.44 −0.29	−0.05 +0.19
					0.67 0.51	0.29 0.29	0.22 0.23	−0.38 −0.22	−0.07 −0.06

TABLE 9

Small Specimen in the Second Row					Large Specimen in the Second Row				
Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$	Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$
1st Row	2nd Row	3rd Row			1st Row	2nd Row	3rd Row		
0.83 0.73	0.49 0.64	0.30 0.49	−0.34 −0.07	−0.19 −0.15	0.72 0.63	0.45 0.45	0.44	−0.27 −0.18	−0.01
0.39 0.52	0.25 0.25	0.23 0.27	−0.14 −0.27	−0.02 +0.02	0.80 0.76	0.47 0.50	0.38 0.51	−0.33 −0.26	−0.09 +0.01
0.37	0.22 0.21	0.15 0.15	−0.15	−0.07 −0.06	0.55 0.82	0.29 0.31	0.19 0.27	−0.26 −0.51	−0.10 −0.04
0.35 0.54	0.22 0.25	0.20 0.16	−0.13 −0.29	−0.02 −0.09	0.44 0.51	0.29 0.28	0.27 0.47	−0.15 −0.23	−0.02 +0.19
0.34	0.20 0.23	0.21 0.23	−0.14	+0.01 0	0.31	0.10 0.18	0.14 0.17	−0.21	+0.04 −0.01
0.28 0.46	0.19 0.16	0.15 0.14	−0.09 −0.30	−0.04 −0.02	0.28 0.45	0.16 0.19	0.20 0.10	−0.12 −0.26	+0.04 −0.09
0.30 0.44	0.19 0.21	0.15 0.16	−0.11 −0.23	−0.04 −0.05	0.32	0.12 0.13	0.19 0.13	−0.20	+0.07 0
0.29 0.64	0.22 0.20	0.16 0.15	−0.07 −0.44	−0.06 −0.05	0.27 0.32	0.10 0.15	0.21 0.12	−0.17 −0.17	+0.11 −0.03
0.34 0.39	0.25 0.30	0.20 0.24	−0.09 −0.09	−0.05 −0.06	0.22	0.18 0.15	0.20 0.19	−0.04	+0.02 +0.04
0.42 0.50	0.30 0.31	0.26 0.22	−0.12 −0.19	−0.04 −0.09	0.70 0.52	0.34 0.27	0.24 0.27	−0.36 −0.25	−0.10 0
0.66	0.62 0.37	0.38 0.41	−0.04	−0.24 +0.04	0.40 0.66	0.26 0.36	0.34 0.38	−0.14 −0.30	+0.08 +0.02
0.36	0.24 0.30	0.21 0.33	−0.12	−0.03 +0.03	0.45 0.54	0.39 0.35	0.40 0.28	−0.06 0.19	+0.01 −0.07

COKE CPR 305 TABLE 10

Small Specimen in the Second Row					Large Specimen in the Second Row				
Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$	Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$
1st Row	2nd Row	3rd Row			1st Row	2nd Row	3rd Row		
0.34 0.50	0.23 0.30	0.24 0.30	−0.11 −0.20	+0.01 0	0.40 0.64	0.30 0.26	0.17 0.21	−0.10 −0.38	−0.13 −0.05
0.50 0.56	0.31 0.23	0.21 0.16	−0.19 −0.33	−0.10 −0.07	0.33 0.44	0.20 0.28	0.16 0.27	−0.13 −0.16	−0.04 −0.01
0.28 0.38	0.15 0.18	0.15 0.10	−0.13 −0.20	0 −0.08	0.31 0.26	0.10 0.15	0.10 0.11	−0.21 −0.11	0 −0.04
0.30 0.29	0.20 0.28	0.11 0.18	−0.10 −0.01	−0.09 −0.10	0.20 0.21	0.20 0.23	0.20 0.19	0 +0.02	0 −0.04
0.38 0.51	0.25 0.22	0.18 0.20	−0.13 −0.29	−0.07 −0.02	1.40 0.70	0.40 0.29	0.29	−1.00 −0.41	−0.11
0.13 0.15	0.13 0.22	0.11	0 +0.07	−0.02	0.43 0.68	0.23 0.30	0.33 0.23	−0.20 −0.38	+0.10 −0.07
0.16 0.37	0.11 0.22	0.14	−0.05 −0.15	+0.03	0.61 0.47	0.27 0.25	0.24 0.26	−0.34 −0.22	−0.03 +0.01
0.20 0.20	0.13 0.14	0.10 0.15	−0.07 −0.06	−0.03 +0.01	1.10 0.89	0.31 0.33	0.23	−0.79 −0.56	−0.08
0.16 0.10	0.16 0.19	0.07 0.09	0 +0.09	−0.09 −0.10	0.83	0.40 0.35	0.31 0.42	−0.43	−0.09 +0.07
0.25 0.32	0.14 0.24	0.11 0.25	−0.11 −0.08	−0.03 +0.01	0.95 1.20	0.41 0.38	0.45 0.33	−0.54 −0.82	+0.04 −0.05
0.20 0.20	0.22 0.14	0.19	+0.02 −0.06	−0.03	0.60 0.55	0.23 0.26	0.32 0.34	−0.37 −0.29	+0.09 +0.08
0.18 0.20	0.18 0.20	0.19	0 0	+0.01	0.58 0.67	0.28 0.36	0.24 0.23	−0.30 −0.31	−0.04 −0.13
0.33 0.58	1.16 0.79	0.60 1.30	+0.83 +0.21	−0.56 +0.51					
0.64 0.62	0.38 0.50	0.60	−0.26 −0.12	+0.22					
0.42 0.41	0.17 0.19	0.35 0.19	−0.25 −0.22	+0.18 0					
0.39 0.75	0.40 0.41	0.62	+0.01 −0.34	+0.21					
0.27 0.25	0.20 0.23		−0.07 −0.02						
0.26 0.27	0.29 0.17		+0.03 −0.10						
0.36 0.36	0.46 0.44		+0.10 +0.08						

TABLE 11

Small Specimen in the Second Row					Large Specimen in the Second Row				
Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$	Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$
1st Row	2nd Row	3rd Row			1st Row	2nd Row	3rd Row		
0.38	0.43 0.28	0.38 0.40	+0.05	+0.05 +0.12		0.55 0.52	0.48 0.59		−0.07 +0.07
0.30	0.21 0.21	0.19 0.14	−0.09	−0.02 −0.07	0.48	0.28 0.44	0.33 0.24	−0.20	+0.05 −0.20
0.27 0.31	0.25 0.21	0.18 0.18	−0.02 −0.10	−0.07 −0.03	0.31	0.16 0.19	0.26 0.23	−0.15	+0.10 +0.04
	0.29 0.32	0.25 0.33		−0.04 +0.01	0.29 0.43	0.17 0.25	0.16 0.20	−0.12 −0.18	−0.01 −0.05
0.22 0.25	0.23 0.23	0.19 0.21	+0.01 −0.02	−0.04 −0.02	0.40	0.26 0.22	0.27 0.26	−0.14	+0.01 +0.04
0.25 0.32	0.22 0.16	0.19 0.23	−0.03 −0.16	−0.03 +0.07	0.20	0.14 0.11	0.17 0.21	−0.06	+0.03 +0.10
0.36 0.60	0.24 0.40	0.31 0.41	−0.12 −0.20	+0.07 +0.01	0.16 0.25	0.10 0.10	0.04 0.05	−0.06 −0.15	−0.06 −0.05
0.28 0.60	0.27 0.28	0.25 0.47	−0.01 −0.32	−0.02 +0.19	0.35	0.25 0.23	0.22 0.30	−0.10	−0.03 +0.07
0.29 0.18	0.18 0.24	0.32 0.35	−0.11 +0.06	+0.14 +0.11	0.36	0.20 0.23	0.22 0.17	−0.16	+0.02 −0.06
	0.30 0.29	0.55 0.38		+0.25 +0.09	0.30	0.14 0.23	0.13 0.14	−0.16	−0.01 −0.07
					0.28 0.34	0.23 0.22	0.21 0.14	−0.05 −0.12	−0.02 −0.08
					0.31 0.25	0.18 0.26	0.24 0.27	−0.13 +0.01	+0.06 +0.01
					0.42 0.41	0.44 0.42	0.90 0.70	+0.02 +0.01	+0.46 +0.28
					0.27 0.23	0.24	0.40 0.30	−0.03	+0.16
					0.08 0.06	0.09 0.17	0.28 0.16	+0.01 +0.11	+0.19 −0.01
					0.10 0.10	0.16 0.16	0.26 0.27	+0.06 +0.06	+0.10 +0.11

TABLE 12

Small Specimen in the Second Row					Large Specimen in the Second Row				
Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$	Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$
1st Row	2nd Row	3rd Row			1st Row	2nd Row	3rd Row		
0.31 0.58	0.35 0.32	0.63 0.52	+0.04 −0.26	+0.28 +0.20	0.17 0.23	0.35 0.65	1.03 0.92	+0.18 +0.42	+0.68 +0.27
0.28	0.52 0.57	0.56 1.00	+0.24	+0.04 +0.43	0.46 0.39	0.37 0.38	0.52 0.45	−0.09 −0.01	+0.15 +0.07
0.61 0.58	0.50 0.76	0.80 0.11	−0.11 +0.18	+0.30 −0.65	0.96	0.53 0.53	0.72 1.04	−0.43	+0.19 +0.51
0.40 0.50	0.34 1.10	0.47 0.56	−0.06 +0.60	+0.13 −0.54	0.53 0.97	0.55 0.78	1.50 1.50	+0.02 −0.19	+0.95 +0.72
0.46	0.29 0.35	0.56 0.46	−0.17	+0.27 +0.11	0.44 1.10	0.32 0.22	0.51 0.40	−0.12 −0.88	+0.19 +0.18
0.32 0.41	0.23 0.43	0.33 0.39	−0.09 +0.02	+0.10 −0.04	0.67	0.48 0.12	0.60 0.61	−0.19	+0.12 +0.49
0.35	0.81 0.90	1 90 1.20	+0.46	+1.09 +0.30	0.63 0.55	0.25 0.35	0.34 0.47	−0.38 −0.20	+0.09 +0.12
0.62 0.73	0.62 0.51	0.96 0.84	0 −0.22	+0.34 +0.33	0.36 0.66	0.23 0.29	0.44 0.64	−0.13 −0.37	+0.21 +0.35
0.77 0.73	0.59 0.38	0.91 1.04	−0.18 −0.35	+0.32 +0.66	0.47	0.22 0.17	0.22 0.40	−0.25	0 +0.23
0.79 0.81	0.50 0.53	0.84 0.74	−0.29 −0.28	+0.34 +0.21	0.17	0.26 0.25	0.24 0.19	+0.09	−0.02 −0.06
0.27 0.43	0.29 0.29	0.45 0.64	+0.02 −0.14	+0.16 +0.35	0.24 0.16	0.15 0.16	0.29 0.32	−0.09 0	+0.14 +0.16
0.40 0.68	0.40 0.42	0.49 0.50	0 −0.26	+0.09 +0.08	0.11 0.15	0.10 0.10	0.20 0.16	−0.01 −0.05	+0.10 +0.06
0.34 0.54	0.33 0.34	0.33 0.31	−0.01 −0.20	0 −0.03	0.22 0.27	0.15 0.25	0.44 0.40	−0.07 −0.02	+0.29 +0.15
0.40 0.18	0.59 0.43	0.63 0.66	+0.19 +0.25	+0.04 +0.23	0.11 0.18	0.10 0.12	0.20 0.20	−0.01 −0.06	+0.10 +0.08
0.32	0.20 0.19	0.40 0.25	−0.12	+0.20 +0.06					
0.22	0.14 0.14	0.23 0.41	−0.08	+0.09 +0.27					
0.20	0.25 0.26	0.37 0.20	+0.05	+0.12 −0.06					
0.14 0.15	0.14	0.24 0.14	0	+0.10					
0.21	0.12 0.12	0.16 0.23	−0.09	+0.04 +0.11					
0.10 0.11	0.12 0.10	0.20 0.23	+0.02 −0.01	+0.08 +0.13					

TABLE 13

Small Specimen in the Second Row					Large Specimen in the Second Row				
Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$	Wt. Gain, In %			$2^R - 1^R$	$3^R - 2^R$
1st Row	2nd Row	3rd Row			1st Row	2nd Row	3rd Row		
0.13 0.15	0.10 0.10	0.22 0.22	−0.03 −0.05	+0.12 +0.12	0.11 0.10	0.10 0.10	0.15 0.13	−0.01 0	+0.05 +0.03
0.24	0.14	0.83	−0.10	+0.69	0.55	0.40	0.60	−0.15	+0.20
0.18	0.16	0.40	−0.02	+0.24					
0.39 0.21	0.19 0.13	0.58 0.25	−0.20 −0.08	+0.39 +0.12	0.22 0.22	0.24 0.14	0.92 0.34	+0.02 −0.08	+0.68 +0.20
0.18 0.15	0.12	0.20 0.52	−0.06	+0.08	0.29 0.23	0.17 0.15	0.33 0.43	−0.12 −0.08	+0.16 +0.28
0.13 0.26	0.15 0.14	0.30 0.34	+0.02 −0.12	+0.15 +0.20	0.20 0.12	0.10 0.10	0.29 0.41	−0.10 −0.02	+0.19 +0.31
0.30	0.26	0.30	−0.04	+0.04	0.12 0.24	0.11 0.10	0.55 0.40	−0.01 −0.14	+0.44 +0.30
0.88	0.41 0.44	0.74	−0.47	+0.33	0.16 0.18	0.17 0.10	0.29 0.35	+0.01 −0.08	+0.12 +0.25
0.50	0.29 0.26	0.49 0.34	−0.21	+0.20 +0.08	0.24 0.22	0.19 0.17	0.55 0.30	−0.05 −0.05	+0.36 +0.13
0.29 0.25	0.22 0.23	0.40 0.43	−0.07 −0.02	+0.18 0.20	0.35 0.25	0.18 0.15	0.24 0.42	−0.17 −0.10	+0.06 +0.27
0.37	0.21 0.22	0.50 0.28	−0.16	+0.29 +0.06	0.20 0.17	0.13 0.12	0.48	−0.07 −0.05	+0.35
0.35 0.25	0.16 0.18	0.40 0.26	−0.19 −0.07	+0.24 +0.06	0.16 0.18	0.11 0.12	0.31 0.24	−0.05 −0.06	+0.20 +0.12
0.40 0.40	0.23	0.32	−0.17		0.22 0.38	0.20 0.20	0.29 0.25	−0.02 −0.18	+0.09 +0.05
					0.25 0.21	0.17 0.16	0.41 0.31	−0.08 −0.05	+0.24 +0.15
					0.35 0.46	0.24 0.24	0.33	−0.11 −0.22	+0.09
					0.79 0.97	0.53 0.82	0.63 0.73	−0.26 −0.15	+0.10 −0.09
					0.43 0.40	0.25 0.30	0.70 0.55	−0.18 −0.10	+0.45 +0.25

TABLE 14

COKE	GAS FLOW	EVAPOR. TEMP	TIME SECONDS	WT. GAIN, IN %		
				1st Row	2nd Row	3rd Row
CPR 296	2.0 l/min	225°C	60	0.52	0.54	0.57
		225°C	60	0.50	0.32	0.32
		225°C	60	0.16	0.20	0.20
CPR 305		227°C	60	0.36	0.32	0.48
		227°C	60	0.43	0.42	0.55
		227°C	60	0.27	0.41	0.93
CPR 306		228°C	60	0.28	0.45	0.57
		227°C	60	0.10	0.20	0.86
		227°C	60	0.33	0.61	0.97
		227°C	60	0.37	0.48	0.68
CPR 307		228°C	60	0.44	0.19	0.11
		230°C	60	0.64	0.64	0.46
		226°C	60	0.41	0.74	0.90
		235°C	60	0.76	0.54	0.38
CPR 308		230°C	60	0.68	0.55	0.34
		230°C	60	0.46	0.30	0.36